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Adhesion Strength of BNT Films Hydrothermally Deposited on Titanium Substrates

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Abstract. Lead-free piezoelectric ($\text{Bi}_{1/2}\text{Na}_{1/2}\text{TiO}_3$) (BNT) films were deposited on 1 mm thick pure titanium(Ti) substrates by a hydrothermal method. Tensile tests were performed to quantitatively assess the adhesion strength between BNT films and Ti substrates. Ti substrates were pretreated by chemical polish and mechanical polish respectively prior to BNT film deposition. In the tensile test, the behavior of BNT film exfoliation was investigated by the replica method. The critical Ti substrate strain inducing BNT film exfoliation was determined by the aid of finite element analysis (FEM). In this study, the results revealed that BNT film exfoliations were caused by the strain of Ti substrate, and the mechanical polish pretreatment improved the adhesion of BNT film to Ti substrate.

Introduction

Piezoelectric materials have been widely used in smart devices such as sensors and actuators. Nowadays, PZT and PZT-based ceramics are the most commonly used piezoelectric materials due to their excellent piezoelectric properties [1,2]. However, lead oxide (PbO) in PZT has a strong toxicity to the human body [3,4]. Hence, there is a great need to develop lead-free piezoelectric ceramics for replacing PZT. In recent years, BNT has been extensively studied as one of promising lead-free piezoelectric ceramics [5]. On the other hand, piezoelectric films have attracted more and more attentions with the great development of MEMS, especially in the development of micro-sensors and micro-actuators [6].

BNT films were successfully synthesized on pure Ti substrates by the hydrothermal method in our previous study. The piezoelectric property of deposited BNT films was confirmed by the measurement of piezoelectric constant (d_{33}) and the actuation test [7]. However, the application and reliability of BNT film/ Ti substrate structures are determined to a large extent by the adhesion of BNT film to Ti substrate. A quantitative evaluation of the adhesion strength of BNT film is demanded in the design and fabrication of BNT film base component. As yet, there is no accepted convention for defining the adhesion strength between a film and substrate [8]. In the present study, tensile tests combined with FEM analysis were conducted to assess the adhesion of deposited BNT films to Ti substrates. Ti substrates were pretreated by chemical polish (CP) and mechanical polish (MP) respectively prior to BNT film deposition with a view of investigating the effects of substrate surface pretreatments on the adhesion strength of BNT films.

Experimental

Sample preparation. BNT films were hydrothermally synthesized on pure Ti substrates which were 1 mm in thickness with a gauge section of 18 mm in length and 6 mm in width. Figure 1(a) shows the dimensions of smooth substrate. In addition, holed substrates, shown in Fig. 1(b), were also prepared

to investigate the effect of stress and strain concentration on the adhesion strength of BNT films. Heat treatment of titanium substrates prior to BNT film deposition was performed in a furnace (KDF-P90G), in an argon inert atmosphere with a heating rate of 10°C /min from room temperature to 700°C. After the heat treatment, some of smooth and holed titanium substrates were mechanically polished by a 500 grit emery paper. The other substrates were chemically polished by immersing them in a chemical solution with the temperature of 90°C. The chemical solution was prepared by nitric acid (HNO₃, 34.3 vol%), hydrofluoric acid (HF, 11.4 vol%) and distilled water. After the pretreatment of mechanical and chemical polish, the substrates were ultrasonically cleaned in a methanol bath followed by a rinse in distilled water. The synthesis process for BNT film was described in our previous paper [7]. BNT film of about 5 µm thick was obtained on each side of Ti substrate.

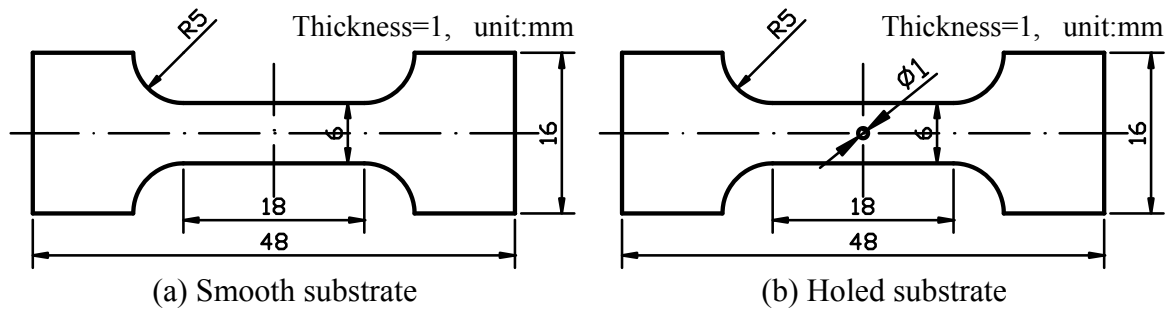


Fig. 1 Schematics of the smooth and holed Ti substrates

Tensile test. The tensile tests were carried out using a universal testing machine (SHIMADZU, AG-100kNG). The stress-strain curve was measured on the heat-treated titanium substrate with the tension speed of 0.02 mm/s up to fracture, with a view of examining the ultimate strength and yield strength of the heat-treated Ti substrate. During the tensile testing of the BNT deposited specimens, the crosshead of the universal testing machine was manually operated to stop at various appointed displacements, and then the behavior of film exfoliation was investigated by the replica method. The film exfoliation would adhere to the replica film, so the behavior of film exfoliation can be investigated by observing the applied replica film. In this study, for the smooth specimen, the number of the film exfoliation in the fixed field of vision was measured by the polarized microscopic observation (OLYMPUS, BX60). For the holed specimen, the behavior of film exfoliation was observed by a stereo microscope (OLYMPUS, SZ1145TR-SPT), and the exfoliation area near the edge of the hole was measured using a laser microscope system (KEYENCE, VK-8510), and then the exfoliation rate was determined by the ratio of the exfoliation area to the gauge section area.

Results and discussion

Smooth specimen. The ultimate strength, yield strength ($\sigma_{0.2}$) and Young's modulus (E) of the heat-treated Ti substrate were determined to be 350 MPa, 195 MPa and 106 GPa respectively from the measured stress-strain curve of the heat-treated Ti substrate. These strength data were used in the FEM model material. In the tensile test of smooth specimens, the applied replica films were observed by the polarized microscopic observation. Figure 2 shows successive observations with the increase of substrate strain for the MP smooth specimen. The critical substrate strain inducing the first film exfoliation was determined to be 1.2% for the MP smooth specimen. Using the above method, the critical substrate strain was determined to be 1.0% for the CP smooth specimen. For both of the MP and CP smooth specimens, the film exfoliation occurred after the yield strength of the Ti substrate, and the number of film exfoliation increased with the increase of substrate strain. Figure 3 shows the variation of the number of film exfoliation with the strain of Ti substrate for these two smooth specimens. The number of film exfoliation increased monotonously with the substrate strain up to 3% after the occurrence of the film exfoliation.

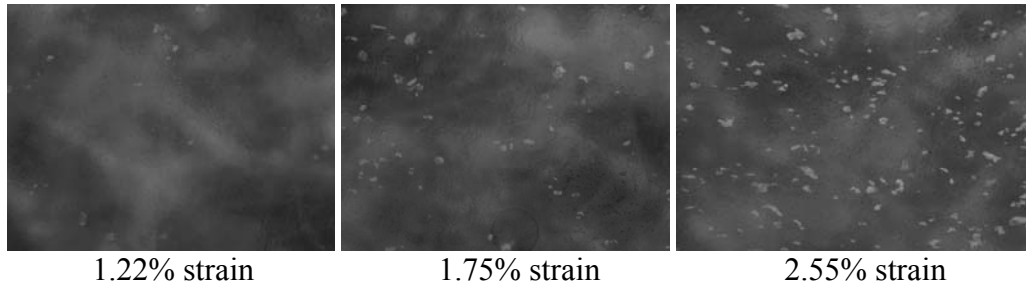


Fig. 2 Successive observations for the MP smooth specimen

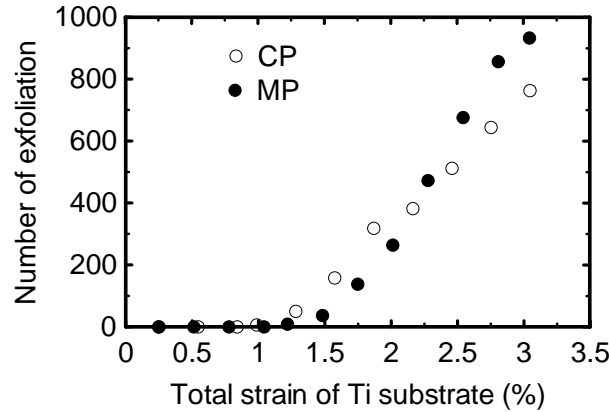


Fig. 3 Variations of the number of film exfoliation with the total strain of Ti substrate for smooth specimens

Holed Specimen. Figure 4 shows successive observations with the increase of strain for the MP holed specimen. The substrate strain in Fig.4 indicates the maximum strain near the edge of hole. The film exfoliation firstly occurred when the substrate strains reached 1.0% and 0.8% for the MP and CP holed specimens, respectively. Figure 5 shows the variations of exfoliation rate with the total strain of Ti substrate for holed specimens. The exfoliation rate of CP specimen increased more largely than that of MP specimen with further increase of substrate strain after the occurrence of film exfoliation. In addition, for both of the MP and CP holed specimens, the film exfoliation expanded in the direction of 45° angle with the tensile direction. The above strains in this section were obtained from the FEM analysis of the holed Ti substrate using ANSYS Res. 11.0 software package. In the FEM analysis, the model material was treated as a homogeneous, isotropic, rate-independent and non-linear plastic body. Figure 6 shows the contrast between microscopic observation and strain contour plot by FEM analysis for the MP holed specimen under the displacement load of 0.26 mm. It can be found that the exfoliation area was in good agreement with the shape of strain contour. By the contrasts of all the results, it was found that the film exfoliation always occurred in the area where the strain was about more than 1.3%.

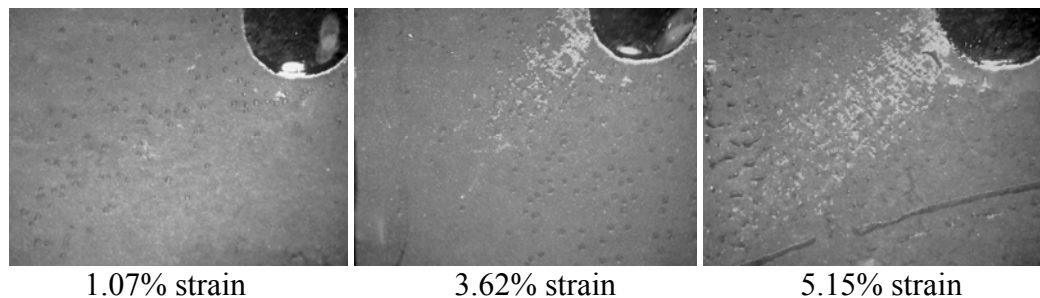


Fig. 4 Successive observations for the MP holed specimen

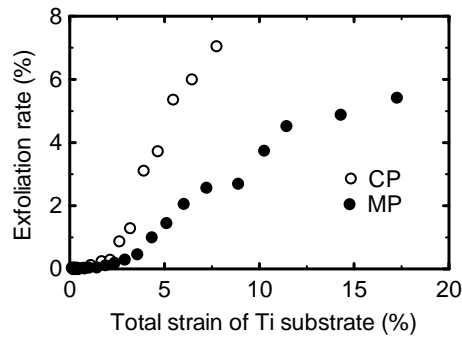


Fig. 5 Variations of exfoliation rate with the total strain of Ti substrate for holed specimens

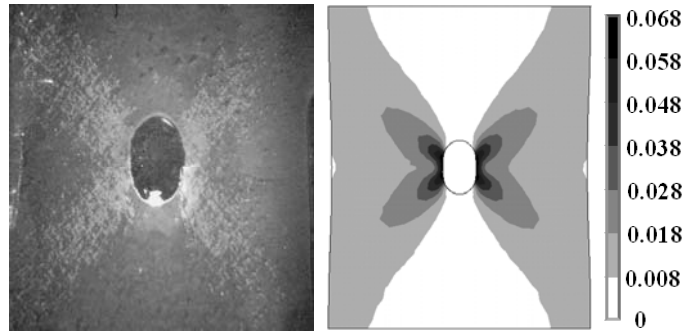


Fig. 6 Contrast between microscopic observation and strain contour plot by FEM analysis

Conclusions

In this study, tensile tests were performed on the smooth and holed specimens to quantitatively evaluate the adhesion strength of deposited BNT films on Ti substrates. The critical substrate strain was obtained with the aid of FEM analysis. The main conclusions are summarized as follows:

- (1) For both of the smooth and holed specimens, the film exfoliation firstly occurred after the yield strength of Ti substrate. The behavior of BNT film exfoliation was attributed to the plastic deformation of Ti substrate.
- (2) The critical substrate strains for the MP and CP specimens were determined to be 1.1% and 0.9%, respectively. This indicates that the pretreatment of the mechanical polish slightly improved the adhesion strength of deposited BNT films, in comparison to the chemical polish pretreatment.
- (3) For the holed specimen, the film exfoliation expanded with increasing the substrate strain in the direction of 45° angle with the tensile direction. The film exfoliation always occurred in the area where the substrate strain was about more than 1.3%. This verified the validity of the obtained critical strain.

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